

WHAT IS CLAIMED IS:

1. A method of generating a supersonic jet expansion, wherein a mixture of a supercritical fluid and a non-volatile sample or a mixture of a supercritical fluid and a pyrolytic sample is jetted into a high vacuum chamber of  $10^{-7}$  Torr or more to generate a supersonic jet expansion of sample molecules in the lowest energy level without intermolecular collisions or molecular aggregates containing the sample molecules.
2. A mass spectrometry method using a supercritical fluid jet method, wherein a mixture of a supercritical fluid and a non-volatile sample or a mixture of a supercritical fluid and a pyrolytic sample is put under high vacuum of  $10^{-7}$  Torr or more to generate a supersonic jet expansion of sample molecules in the lowest energy level without intermolecular collisions or molecular aggregates containing the sample molecules to obtain a molecular beam, ions of the sample molecules in the lowest energy level without intermolecular collisions or molecular aggregates containing the sample molecules are obtained from the molecular beam by performing a laser ionization method, and mass spectrometry is performed on the ions.
3. The mass spectrometry method using the supercritical fluid

jet method according to Claim 2, wherein in a supercritical jet generating device, a pulse valve is used to perform supersonic jetting of a mixture of a supercritical fluid and a non-volatile sample or a mixture of a supercritical fluid and a pyrolytic sample to obtain the supersonic jet expansion, the supersonic jet expansion is introduced via a skimmer into a differential evacuation chamber under a high vacuum of  $10^{-5}$  Torr or more, the supersonic jet expansion is furthermore passed, via a skimmer, through high vacuum of  $10^{-7}$  Torr or more to obtain the molecular beam, the sample molecules obtained from the abovementioned molecular beam or the molecular aggregates containing the sample molecules are ionized from the molecular beam by a resonance-enhanced multiphoton ionization method using a tunable laser, and mass spectrometry is performed on the ions.

4. The mass spectrometry method using the supercritical fluid jet method according to Claim 3, wherein 25 volume % or less of at least one modifier selected from the group of modifiers consisting of water, methanol, ethanol, dioxane, acetonitrile, tetrahydrofuran, diisopropyl ether, and diethyl ether is added to the mixture of the supercritical fluid and the sample.

5. A mass spectrometry device using a supercritical fluid jet method comprising: a supercritical fluid jet generating device that performs supersonic jetting of a mixture of a supercritical fluid and a non-volatile sample or a mixture of a supercritical fluid and a pyrolytic sample; a laser ionization chamber that obtains and ionizes a molecular beam from a supersonic jet expansion jetted from the jet generating device; and a mass analyzer, performing mass spectrometry of ions obtained from the laser ionization chamber.

6. The mass spectrometry device using the supercritical fluid jet method according to Claim 5, wherein a pulse valve that generates the supersonic jet expansion is disposed in the supercritical fluid jet generating device, a differential evacuation chamber is disposed between the jet generating device and the laser ionization chamber, and skimmers are disposed at respective portions through which the supersonic jet expansion passes between the jet generating device and the differential evacuation chamber and between the differential evacuation chamber and the laser ionization chamber.